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(Reaffirmed 1999)

Indian Standard

DETERMINATION OF COBALT IN HARDMETALS BY POTENTIOMETRIC METHOD

1. Scope — This standard covers a potentiometric method for the determination of cobalt content in hardmetals.

1.1 This method is applicable to the following, having cobalt content exceeding 1 percent:

a) Carbides and binder metal powder mixtures free of lubricant, and

b) All grades of presintered or sintered hardmetals.

2. Principle — Oxidation of cobalt to the trivalent state in a strongly ammoniacal solution with an excess of potassium ferricyanide [$K_3Fe(CN)_6$]. Potentiometric back-titration of the excess with cobalt sulphate solution.

3. Interfering Elements

3.1 The effect of interfering elements, for example, vanadium and manganese, shall be taken into account. Vanadium and manganese are also oxidized by potassium ferricyanide. Vanadium reacts stoichiometrically and a correction can be applied if the vanadium content is below 0·5 percent.

3.2 Manganese reacts almost stoichiometrically up to 0·2 percent only if the sum of tantalum and niobium in the hardmetal is less than 0·02 percent. Above this level, the method is applicable only if the manganese content is less than 0·03 percent.

4. Reagents — During the analysis, use only reagents of analytical grade and distilled water or water of equivalent purity.

4.1 *Ammonium Sulphate*

4.2 *Concentrated Hydrochloric Acid* ($\rho = 1{\cdot}19$ g/ml)

4.3 *Hydrofluoric Acid* ($\rho = 1{\cdot}12$ g/ml)

4.4 *Concentrated Nitric Acid* ($\rho = 1{\cdot}42$ g/ml)

4.5 *Concentrated Sulphuric Acid* ($\rho = 1{\cdot}84$ g/ml)

4.6 *Ammonia Solution* ($\rho = 0{\cdot}91$ g/ml)

4.7 *Ammonium Citrate Solution* — Dissolve 100 g citric acid in 900 ml of water and add 100 ml of ammonia solution.

4.8 *Standard Cobalt Solution* (1 litre = 1 g of cobalt) — Prepare the solution from compact metallic cobalt, or from diammonium cobalt sulphate or from cobalt sulphate. If necessary, establish the titre gravimetrically with 1 nitroso-naphthol-2, or electrolytically.

4.9 *Standard Potassium Ferricyanide Solution* (1 litre = 3 g of cobalt) — Dissolve 17 g of potassium ferricyanide in one litre of water.

Determine the relationship between the volume of standard cobalt sulphate solution and potassium ferricyanide solution in the following way.

4.9.1 Transfer into a 400 ml beaker, 5 g ammonium sulphate, 40 ml ammonium citrate solution, 50 to 100 ml water, 80 ml ammonia solution and from a burrette, approximately 9 ml potassium ferricyanide solution. Volume used : V_3 ml.

Immediately titrate with standard cobalt sulphate solution. Volume used : V_4 ml.

Powder Metallurgical Materials and Products Sectional Committee, SMDC 30 [Ref : Doc : SMDC 30 (3013)]

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4.9.2 Calculate the relationship according to the formula:

$$K = \frac{V_4}{V_3}$$

4.10 *Methyl Red Indicator Solution* (1 g/litre), or indicator paper for *p*H 3 to 5.

5. Apparatus

5.0 *Ordinary Laboratory Apparatus*

5.1 *Potentiometric Titration Apparatus* — With stirrer or some other arrangement for agitation.

5.2 *Platinum Electrode*

5.3 *Tungsten Wire or Any Other Reference Electrode*

5.4 *Burrettes* — Of capacity 10 ml.

6. Sampling

6.1 The sample shall be crushed to a powder in a mortar made of a material which does not alter the sample composition. The powder shall pass through a 180 µm sieve.

Note — For sintered hardmetals, the preparation of sample may be as agreed to between the purchaser and the manufacturer.

6.2 The analysis shall be carried out on two or three test portions.

7. Procedure

7.1 Weigh, to the nearest 0·000 1 g, 0·1 to 0·5 g of the test sample, depending on the cobalt content.

7.2 Transfer the test portion into a 400 ml beaker, add 10 to 15 ml concentrated sulphuric acid, 3 ml concentrated hydrochloric acid and 5 g ammonium sulphate. Cover the beaker with a watch-glass and heat until complete dissolution is achieved. Cool. Add, in small portions, 40 to 50 ml ammonium citrate solution. Rinse the watch-glass and the beaker walls with 20 to 50 ml of water. Heat the solution gently until all salts are dissolved. Cool.

7.2.1 The following alternative method for dissolution may be used:

Transfer the test portion into a 400 ml polypropylene beaker or into a platinum dish. Add 10 ml of water and 5 ml of hydrofluoric acid and cover the beaker with a polypropylene cover. Warm to approximately 60°C. Add the concentrated nitric acid, drop by drop, until dissolution is complete. Cool. Add, in small portions, 10 ml concentrated sulphuric acid and 40 to 50 ml ammonium citrate solution (If vanadium is present, heat the solution and cool again). Rinse the cover and the beaker walls with 20 to 50 ml of water.

7.3 Add 1 drop of methyl red indicator solution or use indicator paper. Carefully neutralize the solution with ammonia solution and maintain *p*H 3 to 5 and cool.

7.4 Add 80 ml of ammonia solution and preferably cool to 10°C. Immediately add potassium ferricyanide solution from the burrette to an excess of a few millilitres. Volume used : V_1 ml.

Back titrate with a standard cobalt sulphate solution. Volume used : V_2 ml.

8. Test Results

8.1 *Calculation* — The cobalt content, expressed as a percentage by mass, is given by the formula

$$\text{Cobalt content (percent by mass)} = \frac{K \times (V_1 - V_2) \times T \times 100}{m}$$

where

K = defined in **4.9.2**;

T = mass, in g, of cobalt in 1 ml of the cobalt sulphate solution; and

m = mass, in g, of the test portion.

8.1.1 The result shall be rounded to the nearest 0·01 percent.

8.2 *Corrections* — The following correction values may be used:

Vanadium : 0·100 percent vanadium is equal to 0·116 percent cobalt, and
Manganese : 0·100 percent manganese is equal to 0·107 percent cobalt.

8.3 *Tolerances* — The deviations between two or three independent determinations shall not exceed the values shown in the Table 1.

TABLE 1 TOLERANCES

Cobalt Content	Range for Two Determinations	Range for Three Determinations
Percent	Percent	Percent
From 1 to 5	0·10	0·12
Over 5 to 8	0·15	0·18
Over 8 to 20	0·20	0·25
Over 20	0·30	0·35

8.4 *Final Result* — Report the arithmetical mean of acceptable determinations rounded to the nearest 0·1 percent.

9. Test Report — The test report shall include the following information:

a) Reference to this standard,
b) All details necessary for identification of the test sample,
c) The result obtained, and
d) Details of any occurrence which may have affected the result.

EXPLANATORY NOTE

In the preparation of this standard, assistance has been derived from ISO 3909-1976 'Hardmetals — Determination of cobalt — Potentiometric method', issued by the International Organization for Standardization (ISO).

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